PRESENTATION TO

MASSACHUSETTS DEPARTMENT OF ENVIRONMENTAL PROTECTION

FROM: OLIN CORPORATION

RE: RESPONSIBILITY OF AMERICAN BILTRITE, INC.

September 23, 1994

AMERICAN BILTRITE INC.

CONTACT:

Henry W. Winkleman, Esq. Corporate Secretary, Corporation Counsel & Tax Manager

ADDRESS:

American Biltrite Inc. 57 River Street Wellesley Hills, Massachusetts 02181-2097

TELEPHONE:

(617) 237-6655

RESPONSIBILITY OF AMERICAN BILTRITE INC. FOR CONTAMINATION AT THE OLIN WILMINGTON SITE

Olin Corporation, the current owner of the former chemical manufacturing plant located at 51 Eames Street, Wilmington, Massachusetts, has been performing alone the cleanup activities necessitated by the waste disposal practices of several former owners and operators. Olin acquired the plant in 1980 and conducted operations there until 1986. Olin has cooperated with the Department of Environmental Protection in conducting needed studies and cleanup of contamination associated with the facility. Olin recently submitted to the DEP its signed Transition Statement for the site, in order to move forward with further necessary work.

Now Olin requests that a former owner and operator, American Biltrite Inc., be issued a Transition Statement and requested to sign as a co-permittee for the work remaining to be performed at the site. It is clear from the data collected over the past few years that former owners and operators of the Wilmington plant, from the early 1950s through the 1960s, are primarily responsible for the contamination of the site because of both their waste disposal practices and the nature of chemicals used in their plant operations.

First, until approximately 1971, virtually all plant wastes were dumped untreated into unlined pits and lagoons onsite; in about 1971, a wastewater treatment system, with lined lagoons, was constructed at the plant and treated wastewaters were sent off site through a publicly owned treatment works. Second, chromium, a key contaminant of concern in the deep groundwater plume and other areas of the site, was used in a major plant process -- and discharged directly into the unlined pits and lagoons -- only until 1967, but not after that time. Thus, while chromium is a significant contaminant at the site, the use and discharge of chromium was eliminated more than a decade before Olin acquired the plant.

The former owners/operators who are primarily responsible for site contamination include American Biltrite Inc. ("ABI"), The Biltrite Corporation, Fisons plc, and NOR-AM Chemical Company. These former owners and operators have refused to assist in the cleanup, despite the fact that they have been issued Notices of Responsibility for the site contamination and have been repeatedly asked by Olin, pursuant to Section 4A of Chapter 21E, to assist and participate. Because of these parties' refusal to participate in the cleanup, Olin has filed suit in federal court. 1

Olin acquired the plant in 1980 from Stepan Chemical Company. Stepan purchased the plant in 1968 from a British company, Fisons (continued...)

ABI is a documented owner and operator of the site that, for a decade or more, was responsible for the disposal of untreated wastewaters, including wastes containing chromium, into unlined pits and lagoons. Indeed, ABI is responsible for a majority of the chromium and other contamination at the site. It is unfair for Olin to continue to bear the sole burden of work that can, and should, be done by ABI.

Although Olin's investigation is ongoing, documents found to date, interviews with former plant employees, and other materials show that ABI's responsibility for site contamination is clear cut. Facts regarding ABI's responsibility are set forth below.

I. PRE-1970 WASTE DISPOSAL PRACTICES:

From the time of the plant's construction in the early 1950s and through most of the 1960s, untreated plant wastes were disposed in unlined waste disposal pits located south of plant manufacturing buildings and in an unlined lagoon on the west side of the plant known as "Lake Poly."

This statement is based on the following facts:

- 1. Former plant employees including former plant manager Charles P. Riley, Jr. -- whose affidavit is attached -- stated that untreated liquid wastes were dumped into the ground through such sources as unlined pits and lagoons. (Exhibit 1, ¶ 3). Two unlined pits were located on the southern side of the plant buildings and the large unlined lagoon, known as Lake Poly, was on the western side of the plant buildings. Stepan changed these waste disposal practices shortly after it acquired the plant in 1968, by eliminating Lake Poly and constructing a wastewater treatment system including lined lagoons.
- 2. Aerial photographs confirm that Lake Poly was installed by 1954, along with the construction of the first plant buildings. Lake Poly does not appear in a 1952 photo (Exhibit 2), but in a 1954 photo, Lake Poly is even larger than the plant buildings (Exhibit 3). The 1954 photo also shows what appears to be a second man-made oily looking pond

settlement with Olin to share a portion of the costs for cleanup.

U...continued)
Ltd. (now Fisons plc), and one of its U.S. subsidiaries. Stepan did not use chromium, built a wastewater treatment plant on site, and discontinued the prior practice of dumping wastes into unlined pits and lagoons. Stepan has already entered a private

further south of Lake Poly. A photo from approximately 1963 (Exhibit 4) shows that Lake Poly continued in existence then, along with the disposal pits.

3. A 1963 memorandum written by a representative of Fisons Ltd. when that company was considering buying the plant from ABI, says that "[t]here is no effluent treatment system whatever: all waste liquors are piped to an adjacent field and dumped in a hole." (Exhibit 5, p. 1).

II. THE CHROMIUM PROCESS WAS USED ONLY IN THE 1950s AND 1960s:

As the DEP is aware, chromium is a key contaminant of concern in the deep groundwater plume. Chromium is also found in the flocculent that has occurred in the west ditch and in ditch sediments and soils around the site. In addition, most of the current site work is related to chromium contamination. However, chromium contamination was not caused by Olin and could only have been caused by the former owners and operators, especially ABI.

As explained below, from the mid-1950s until 1967, chromium was used in the production of Kempore, a chemical blowing agent that was a major product of the plant. Wastes from this process containing chromium were disposed in the unlined pits and Lake Poly. But the use of chromium in the Kempore process ended in 1967, long before Olin acquired the plant in 1980.

This statement is based on the following facts:

- 1. Former employees, including Charles Riley, recall that chromium was used in the Kempore (azodicarbonamide) process beginning in the mid-1950s and ending in 1967 (Exhibit 1, ¶¶ 2-4).2/
- 2. Production records from 1963 (Exhibit 7) show that bichromate continued to be used in the Kempore process in 1963. 3

A disclosure statement (Exhibit 6, p. 2) Olin obtained when it purchased the plant states that Kempore production using the chromium-based oxidation process began in 1956.

Olin has located to date only a few actual production records from the 1950s and 1960s, although we have requested the prior owners and operators to provide any such documents they have. Olin has found daily production records only for the period August through December 1963. Exhibit 7 includes portions of several of these records as examples to show the continuing use (continued...)

- 3. Charles Riley and other former plant employees who worked directly with the Kempore process confirmed that chromium wastewaters were dumped in the unlined pits (Exhibit 1, ¶ 3). In the chromium Kempore process, sodium bichromate was added and the product went through a washing process. The wash water was dumped into the unlined pits on site.
- 4. Charles Riley, who was plant manager in 1967, recalls that the use of sodium bichromate ended in 1967, when a new chlorate-based process was installed (Exhibit 1, ¶ 4). This process switch is also confirmed by plant files dating from 1967 (Exhibit 8).
- 5. The fact that the chromium process was used by only earlier plant operators is also supported by the patent rights that the various companies obtained. While American Biltrite and Fisons both obtained rights to use the patent for the chromium-based Kempore (azodicarbonamide) process, patent #2,988,545, Stepan obtained a license giving it the right to use the patent for the chlorate-based process, #3,366,622 (Exhibit 9).
- 6. After the chromium-based process was discontinued in 1967, it was never resumed at the plant under either Stepan or Olin. This is based on the statements of former plant employees who remained at the plant until it was shut down in 1986. This information is also confirmed by a review of Kempore operating procedures post-1967 and until plant closure (Exhibit 10).5/

J(...continued)
of the chromium-based oxidation process. Reports covering a
total of 84 days of production from August 27 to December 31,
1963 are available upon request.

Because of the volume of the files involved, only a few documents are included in the exhibit. A full set of the documents that Olin has relating to the change in the Kempore process is available upon request.

Portions of operating procedures are included in the exhibit as examples because of the volume of documents involved. A full set of operating procedures that have been found is available upon request.

III. ABI IS RESPONSIBLE FOR MOST OF THE SITE CONTAMINATION:

ABI is responsible for all plant operations and waste disposal from the plant's construction in the early 1950s until February 1964. ABI is responsible for most of the chromium wastes and other contaminants that were dumped in unlined pits and lagoons at the site.

This statement is based on the following facts:

- 1. The plant was initially operated under the name of National Polychemicals, Inc. ("NPI"). NPI's parent company, American Biltrite Rubber Co. ("ABR") liquidated NPI and took all its assets in January 1964. (Exhibit 11).
- 2. When it liquidated NPI, <u>ABR agreed to accept all of the liabilities of NPI</u>. This agreement is contained in January 24, 1964 Bill of Sale and General Conveyance between ABR and NPI (Exhibit 12, pp. 1, 4-5).
- 3. After liquidating NPI, ABR directly owned and operated the plant until February 17, 1964, when it conveyed the plant to a new subsidiary of Fisons Ltd. This is shown by the January 24, 1964 Bill of Sale (Exhibit 12) and the Instrument of General Transfer in which ABR transferred the plant and other NPI assets. (Exhibit 13).
- 4. In 1973, ABR changed its name to American Biltrite Inc., as shown by a restated Certificate of Incorporation (Exhibit 14).

IV. RESPONSIBILITY OF OTHER PARTIES:

Olin believes several other former owners and operators share responsibility as well, including: The Biltrite Corporation, Fisons plc, and NOR-AM Chemical Company. However, the different factual and legal issues regarding the other parties' liability requires further investigation at this time. As to these parties, Olin asks the assistance of DEP in the form of requests for information under Chapter 21E section 8. Requests for information would force the other parties to disclose important facts that they have withheld during discussions pursuant to section 4A of Chapter 21E regarding prior plant operations, sources of contamination, and their own responsibility for contamination. For example, additional information on wastes generated and disposal practices used throughout the plant's history, the burial of drums on-site, and the presence of pesticides at the site would assist the remaining site characterization work. This information would also help Olin to obtain the participation of these responsible parties in the cleanup process.

CONCLUSION

Olin remains committed to working with the DEP in a responsible manner to accomplish the cleanup of its Wilmington facility. However, Olin should not have to conduct the cleanup alone while ABI, and the others that contaminated the plant, ignore their responsibility.

Because ABI's responsibility for the bulk of the contamination is clear, ABI should be required to participate in the cleanup effort. It has been informed of the site conditions and the status of the ongoing work by Olin and by the DEP. ABI representatives have been on-site to view current conditions. Olin informed ABI of the reclassification of the site as a Tier IA priority site and has provided ABI with many background documents such as the Comprehensive Site Assessment documents. Olin has also made available the Work Plan for supplemental Phase II field work that has been approved by the Department and has invited ABI to participate in this work.

Because ABI is a responsible party under Chapter 21E and, in fact, caused most of the current contamination associated with the facility, ABI should be required to participate in the site cleanup. Olin requests that ABI be issued a Transition Statement and requested to sign as a co-permittee for the work remaining to be performed at the site.

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DECLARATION OF CHARLES P. RILEY, JR.

CHARLES P. RILEY, JR., an adult citizen of the State of Illinois, states as follows:

- 1. I was employed at a chemical manufacturing plant at 51 Eames Street, Wilmington, Massachusetts (the "plant"), from 1957 until 1976. I was in the Research Department for about 10 years. I was Chemical Development Manager in that department from 1964 until January 1, 1967. As Chemical Development Manager, I was responsible for developing chemical processes at the plant and had knowledge of all production processes in use at the plant and plans for developing new processes. I became the Plant Manager on January 1, 1967 and held that position until June 1, 1969. In that position, I was responsible for oversight of plant production activities. Thereafter, I was General Manager with overall responsibility for the plant until I left Wilmington in 1976.
- 2. When I came to work at the plant, Kempore was one of its major products. Kempore is the trade name for azodicarbonamide, a chemical "blowing agent" used in the rubber and plastics industries. Blowing agents create pockets of air to give rubber and plastic products greater buoyancy and flexibility. Kempore remained a major product throughout my employment at the plaint and, even before I was hired, I understand that Kempore had been produced from about the mid-1950s.

- 3. For a period of about 10 years after I was hired at the plant, a chromium-based oxidation process was used to make Kempore. In that process, a chromium compound (sodium bichromate) was introduced to a batch prior to final washing. The waste washwater, which contained chromium, was disposed of on-site by dumping into unlined pits. Unlined pits and lagoons were used until the early 1970s to dispose of wastes from each of the chemical manufacturing operations at the plant, including the Kempore process. I was told that the same unlined disposal pits and lagoons used during my tenure had been used for waste disposal virtually from the plant's inception.
- 4. The chromium-based oxidation process for Kempore was used until the spring of 1967. At that time, a chlorine-based oxidation process was instituted. At that point, use of sodium bichromate ceased and chromium no longer was a constituent of the waste stream at the plant, including the waste washwater.
- 5. In the early 1970s, shortly after the acquisition of the plant by the Stepan Chemical Company, a wastewater treatment facility, including lined lagoons, was constructed. All liquid wastes from the manufacturing plants were treated and the treated water went off site through a sewer. Solid wastes settled out in the lined lagoons and formed a gypsum "cake," which was periodically deposited in an on-site landfill.
- 6. I make this declaration on the basis of my own personal knowledge, unless otherwise indicated.

I declare under penalty of perjury that the foregoing is true and correct.

Executed on: September 21 /994

Charles P. Riley, JE.

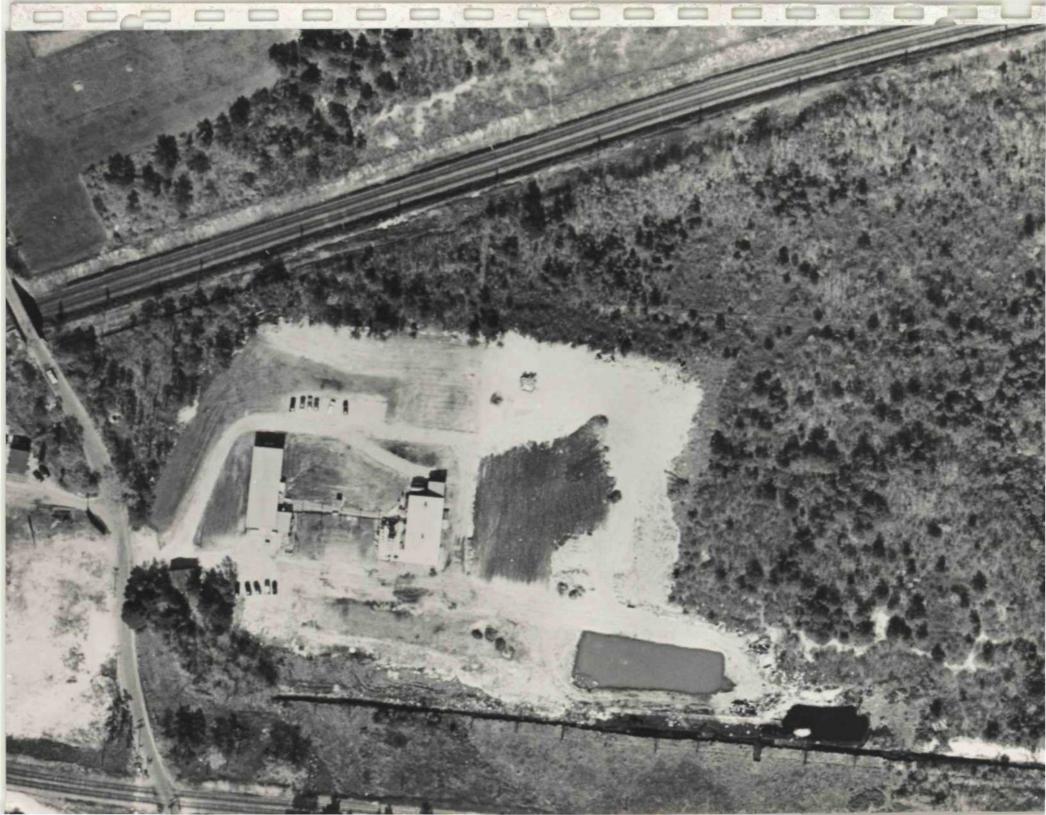
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DISPOSAL PITS



STEPAN CHEMICAL COMPANY/OLIN CORPORATION AGREEMENT OF PURCHASE AND SALE

AGREEMENT made as of the 5th day of August, 1980, by and between STEPAN CHEMICAL COMPANY, a Delaware corporation with principal offices in Northfield, Illinois ("Seller"), and OLIN CORPORATION, a Virginia corporation with principal offices at 120 Long Ridge Road, Stamford, Connecticut ("Buyer").

WITNESSETH:

whereas, Seller is the owner of facilities and other assets and properties for the manufacture of chemical blowing agents and other related chemicals located at Wilmington, Massachusetts known as the Polychem Division of Stepan Chemical Company ("Polychem"); and

WHEREAS, Seller desires to sell, and Buyer desires to purchase all of the assets, properties, goodwill and business of Polychea as a going concern including the assets and properties set forth in Section 1.02 below subject to and in accordance with the terms and conditions hereinafter set forth;

NOW, THEREFORE, in consideration of the respective representations, warranties, covenants and agreements contained herein, Buyer and Seller hereby agree as follows:

3.06 Disclosure

Sits

in a joint meeting on April 30, 1980 at the Boston office of Sullivan and mister, representatives of Olin, Stepan, the Attoney General's office, and the conformal of Hater Pollution Control discussed the disposition of the above court in It was agreed between the representative of the Attorney General and the mismatrive of the Division of Hater Pollution Control that if Olin Corporation in the Stepan, Hilmington plant site, the following would apply:

- 2.) Olin would have 12-18 months for an engineering study to define pollution problems and to suggest remedies.
- :.) There would be no prosecution by the State during this period.
- :.) Olin could expect to expand operations at the site.

The Attorney General and Stepan's counsel have jointly applied for a two month retion of the court action to accomodate Olin's decision on purchase of the larty.

The seller has no knowledge of any other suit, threatened suit, actions, state or local law or other transental matters.

Thrufacturing Processes Conducted on Hilmington Site, Sellers' Best Knowledge

a.) Opex - (Dinitrosopentamethylenetetramine)

Raw materials: Hexamethylenetetramine

Sodium nitrete nitrite

Hydrochloric acid

Processing oil - Ammonia

Product: Diaitrosopentamethylenetetramine (solid)

Buildreducts in waste: Sodium chloride

Sodium nitrite nitrate

forma luchyde

Processing oil - Annionium Chloride

Process operated: 1953-1980

"All information prior to Stepan's purchase of plant in 1968 is based on knowledge of Ron McBrien and Charles P. Riley, Jr. Stepan has not made any investigation with regard to matters before it acquired the plant in 1968.

Erhibit 3.06 invironmental Disclosure

b.) Kempore - (Azodicarbonamide)

Raw materials: Hydrazine

Urea

Sulfuric acid

Sodium chlorate

Sodium BromideBromine (catalyst level)

Product: Azodicarbonamide (solid)

By-products in waste: Sodium sulfate

Sulfuric acid

Urea

Sodium chloride Ammonium sulfate

Sodium Bromide

Un to 1967, sodium dichromate was used in the process instead of Sodium chlorate. The by-product waste contained chrome sulfate instead of sodium chloride and sodium sulfate.

Process operated: 1956-1980

c.) Hydrazine

Raw materials: Ures

Chlorine

Sodium hydroxide Sulfuric acid

Product: Hydrazine and Semicarbazide solution

By-products in waste: Sodium sulfate

Sodium chloride

Ammonium Sulfate

Note: This process was shut down in the fall of 1970 with hydrazine then purchased from Olin Corporation. (operated 1963-1970)

d.) Kempore Dispersions

Raw materials: Azodicarbonamide

Dioctyl phthalate

Product: 50 percent dispersion of Azodicarbonamide in Dioctyl Phthalate

Bisproducts in waste: None

Process operated: 1960-1980

e.) Wytox 312 - (trisnonylphenyl phosphite)

Raw materials: Nonyl phenol

Phorphorus trichloride

Product: Trisnonylphenyl phosphite (liquid)

By-products in weste: None (hydrogen chloride scrubbed in water)

Process operated: 1965-1980

'ranibit 3.06 Environmental Disclosure

f.) Actafoem R-3

Raw materials: 2-ethylhexoic acid

Zinc oxide

Dioctyl phthalate

Sodium oleate Potassium oleate

Product: Liquid Azodicarbonamide activator

By-products in waste: None Process operated: 1963-1980

g.) Hytox Pap - (Alkylated phenol)

Raw materials: Nonyl phenol

Dinonyl phenol Formaldehyde

Product: Liquid Alkylated phenol

By-products in waste: None Process operated: 1971-1980

h.) <u>Nitropore 5PT</u> - (5-phenyltetrazole)

Raw materials: Benzonitrile

Sodium azide Sodium nitrite Ammonium chloride Hydrochloric acid

Product: 5-phenyltetrazole (solid)

By-products in weste: Sodium chloride

Sodium nitrate Be

Dimethyl Formamide

Dimethyl Formamide

Benzonitrile

(produced 1973-1980, limited quantities 24,000 lbs./yr.)

i.) Nitropo<u>re OT - (4.4' oxybisbenzenedisulfonylhydrazide)</u>

Raw magerials: Denhenyl oxide

Chlorosulfonic acid

Hadrezine

Ammonia

auxi · whill).

Product: 4,4' exybisbenzenedisulfonylhydrazide

By-products in weste: Sulfuric acid

Hydrochinric-seld Ammonium Chloride

Process operated: 1969-1975 (Hydrochlaric acid scrubbed in water

j.) Mytox ADP - (Dioctyl Diphenylamine)

Raw materials: Diphenylamine

Dissobutylene

Aluminum hydroxide

-Sedium-chieride

Sodium hydroxide

Aluminum chloride

OW 014649

itit 3.06

Product: Dioctyldiphenylamine

By-products in waste: Aluminum hydroxide

Sodium chioride Sodium hydraxide

Process terminated in 1971. (Operated 1962-1971)

k.) Phenolic and Urea Formaldehyde Resins

Raw Materials: Phenol

Urea

Formal dehyde

Products: Solid and liquid resins

By-products in waste: Phenol

Formal dehyde

Process operated 1961-1967

1.) Phthalate Plasticizers - (dioctyl phthalate, dibutyl phthalate)

Raw materials: Phthalic anhydride

2-ethylhexanol Butyl alcohol

Products: Liquid plasticizers

By-products in waste: None Process operated: 1955-1961

m.) Hiltrol N - (N-Nitrosodiphenylamine)

Raw materials: Diphenylamine

Sodium nitrite Sulfuric acid

Product: N-Nitrosodiphenylamine (solid)

By-products in waste: Sodium nitrite

Sodium sulfate

Process operated: 1965-1967

J. Chemical Waste Disposal

To the seller thest knowledge, chemical manufacturing commenced on the Wilmington plant site around 183. He waste treatment facility or sewer was included in the original plant factities. The main plant effluents were discharged to a small mannade pond on the west side of the property which flowed to a brook bisecting the property. The brook exited the property at the east side along the railroad right of way.

Stromm acid wastes were conducted separately to unlined man-made lagoons on the south side of the operating plant buildings.

* Small quantities of miscellaneous materials were manufactured at various times which had no by-product wastes.

July Du, 1980 Page Live

rehibit 3.06 Invironmental Disclosure

In 1969, Stepan Chemical Company, Inc. and the Division of Water Pollution Control of the Commonwealth of Massachusetts entered into a consent order whereby Stepan was to build a waste treatment plant and the effluents from the treatment plant would be discharged to a Metropolitan District Commission sewer which was to be extended to the immediate area of the plant. Stepan was allowed to continue to discharge chemical wastes in the same manner until the treatment plant was completed. In 1970, with the completion of the treatment plant construction, Stepan was requested to operate the plant and discharge the treated wastes through its property until the sewer construction complete. The sewer was finished in 1972 and approval was granted to Stepan to discharge the effluents from the treatment plant into the sewer by the Metropolitan District Commission.

The Stepan treatment facility neutralizes sulfuric acid and sulfate wastes with a lime system. The resulting calcium sulfate by-product is separated from the effluent by settling in vinyl lined languages. The Department of Public Health of the Commonwealth of Massachusetts approved the construction of a landfill on the Stepan property for the disposal of the calcium sulfate by-products.

The seller has no knowledge of chemical or other dumping on the plant site during its ownership by its own personnel or third parties except as described previously with the discharge of operating wastes. The seller has no knowledge of chemical or other dumping on the property in prior periods.

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UHEA: 6500 V	
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DAKAD: 20 V	
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DRUMS (N32 YELLOW):	
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DRUMS (W55 FIBER):	2
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SILICA:	48000
ICE:	66
DRUMS (N32 CLEAR):	44'
PINISHED PRODUCT:	8800
KENPORE	
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SULFURIC ACID:	8104
DAXAD:	13
RICHROKATE:	4000
ANTIPOAN:	600
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DRUMS (W55 FIBER):	4
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BOXES AND LINERS:	
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FINISHED PRODUCT (TYPE & AMOUNT) Kemps	ou 125 5750
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PORT:	9900
SALe	13500
BAXAD:	5
MURIATIC ACID:	60301
598 OIL:	360 V
SILICA:	4800
ICB:	61 V
DRUMS (N32 CLEAR):	43
PINISHED PRODUCT:	8600
KENPORK	
URBA:	6500
MONORYDRAZ INE:	4830
SULFURIC ACID:	10.335
DAXADa	20
BICHROMATE:	5200
ANTIFOAM:	8
tota Plant Hydreine 1200 cal	1486
DRUMS (W55 FIBER):	2
DRUMS (N32 YELLOW):	40
BOXES AND LINERS:	
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0.40M3 (M55 11 ER PT	Plant Hydrosin: 3600 Gals	3922
ECCENTRIC CONTROL OF CITY A. S. ASOUNT): Campus 125 (375)	UNUMA (W55 FIELD):	3
FINISHED PROBLET (IY & & AFOUNT): Company 125 6375	(DMS (N32 Yelliam):	51/
PANISHED PROBLET (19 1. & AFOUNT): Kempne 125 6375	BUNES For Alberton	
PANISHED PROBLET (19 1. & AFOUNT): Kempne 125 6375	KENNULL 200	450
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	Nov. 29 1963
PRODUCT A	LBS.
PORT:	3300 /
SAL:	4500
DAXAD:	
MURIATIC ACID:	2010
598 OIL:	120
SILICA:	1200
ICE:	23
DRUMS (N-32 CLEAR):	11
FINISHED PRODUCT:	2200
KEMPORE	
UHEA:	3300
Monohydrazine:	2415
SULFURIC ACID:	6120
DAXAD:	100
BICHROMATE:	3000 ;
ANTIFOAM:	2 1
Box: Plant Hydrozin 1200	1156
DRUMS (W-55 FIBER):	
DRUMS (N-32 YELLOW):	
BOXES AND LINERS:	
KEMPORE 200:	
FINISHED PRODUCT (TYPE AND RMOUNT):	
	,

	Dec. 2 1963
PRODUCT A	LBS.
PORT:	
SALs	
DAXAD:	
MURIATIC ACID:	
598 OIL:	
SILICA:	100
ICE:	
DHUMS (N-32 CLEAR):	
FINISHED PRODUCT:	
CENT-OFIE	
UHEA:	8200
MONCHYDRAZINE:	6040 /
SULFURIC ACID:	14512 / 1
DAXAD:	20/
BICHROMATE:	6900/
Antipoam:	2/
see: Plant Hydrogina 2550 Gols	2377
DRUMS (W-55 FIBER):	
DRUMS (N-32 TELLOW):	
BOXES AND LINERS:	
KEMPONE 200:	
FINISHED PRODUCT (TYPE AND MACUNT): Kampore 6	0 2625
72	

	Lec. 5 1763
PRODUCT A	LBS.
PORT:	99000
SAL:	13500
DAXAD:	5/
MURIATIC ACID:	6030/
598 OIL:	360
SILICA:	4800/
ICE:	53/
DRUMS (N-32 CLEAR):	44
FINISHED PRODUCT:	8500
KEMPORE	/
UREA:	6 6 00'
MONOHYDRAZINE:	48301
SULFURIC ACID:	9736
DAXAD:	9736V 15V
BICHROMATE:	4900 :
ANTIFOAM:	2
100. Plant Hydragin 1200	1191
DRUMS (W-55 FIBER):	4
DRUMS (N-32 YELLOW):	62
BOXES AND LINERS:	
KEMPORE 200:	600
FINISHED PRODUCT (TYPE AND RMOUNT): Kemps	u 125 7750
	

MURIATIC ACID: 598 OIL: SILICA: ICE: DRUMS (N-32 CLEAR): FINISHED PRODUCT: EMPORE UREA: MONOHYDRAZINE:	Lae. 9, 1963 LBS. 19800 27,000 27,000 720 9000 126 82 16900 16300 12075 24578 40 12500
PORT: SAL: DAXAD: MURIATIC ACID: 598 OIL: SILICA: ICE: DRUMS (N-32 CLEAR): FINISHED PRODUCT: EMPORE UREA: MONOHYDRAZINE: SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: DRUMS (W-55 FIBER):	12060 7200 7200 9000 126 820 16 900 12 075 24 578 40
MURIATIC ACID: 598 OIL: SILICA: ICE: DRUMS (N-32 CLEAR): FINISHED PRODUCT: EMPORE UREA: MONOHYDRAZINE: SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: DRUMS (W-55 FIBER):	12060 720 9000 126 82 16 400 16 300 12 075 24 578 40
MURIATIC ACID: 598 OIL: SILICA: ICE: DRUMS (N-32 CLEAR): FINISHED PRODUCT: EMPORE UREA: MONOHYDRAZINE: SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: DRUMS (W-55 FIBER):	12060 720 9000 126 82 16 400 16 300 12 075 24 578 40
MURIATIC ACID: 598 OIL: SILICA: ICE: DRUMS (N-32 CLEAR): FINISHED PRODUCT: EMPORE UREA: MONOHYDRAZINE: SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: DRUMS (W-55 FIBER):	12060 720 9000 126 82 16 400 16 300 12 075 24 578 40
598 OIL: SILICA: ICE: DRUMS (N-32 CLEAR): FINISHED PRODUCT: EMPORE UREA: MONOHYDRAZINE: SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: MONOHYDRAZINE: ANTIFOAM: GRUMS (W-55 FIBER):	720 V 9000 / 126 / 82 V 16 400 16 300 12 075 V 24 578 V 40 /
SILICA: ICE: DRUMS (N-32 CLEAR): FINISHED PRODUCT: EMPORE UREA: MONCHYDRAZINE: SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: DRUMS (W-55 FIBER):	9000 126 82 16 900 16 300 12 075 24 578 40
ICE: DRUMS (N-32 CLEAR): FINISHED PRODUCT: EMPORE UREA: MONCHYDRAZINE: SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: DRUMS (W-55 FIBER):	126 82 16 400 16 300 12 075 24 578 40
DRUMS (N-32 CLEAR): FINISHED PRODUCT: EMPORE UREA: MONOHYDRAZINE: SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: JONE Plant Hydrogine 3600 cale DRUMS (W-55 FIBER):	82 V 16 400 16 300 12 075 V 24 578 V 40 V
FINISHED PRODUCT: EMPORE UREA: MONOHYDRAZINE: SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: JOHN Plant Hydrogine 3600 cale DRUMS (W-55 FIBER):	16 400 16 300 12 075 24 578 40
UREA: MONCHYDRAZINE: SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: JEF: Plant Hydrogine 3600 Fals DRUMS (W-55 FIBER):	/6 300 /2 075 24 578 40
UREA: MONOHYDRAZINE: SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: SEP: Plant Hydrogine 3600 Fals DRUMS (W-55 FIBER):	12 075 V 24 578 V 40 V
MONOHYDRAZINE: SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: SEP: Plant Hydrogine 3600 Fals DRUMS (W-55 FIBER):	12 075 V 24 578 V 40 V
SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: SEP: Plant Hydrogine 3600 Eals DRUMS (W-55 FIBER):	12 075 V 24 578 V 40 V
SULFURIC ACID: DAXAD: BICHROMATE: ANTIFOAM: SEP: Plant Hydrogine 3600 cals DRUMS (W-55 FIBER):	24,578
DAXAD: BICHROMATE: ANTIFOAM: SEP: Plant Hydrogine 3600 Fals DRUMS (W-55 FIBER):	40/
ANTIFOAM: 687: Plant Hydrogine 3600 cals DRUMS (W-55 FIBER):	12 500
DRUMS (W-55 FIBER):	/ ~ ~ ~ /
DRUMS (W-55 FIBER):	4 /
DRUMS (W-55 FIBER):	3253
DRUMS (N-32 YELLOW):	2
	19.
BOXES AND LINERS:	
KEMPORE 200:	300
FINISHED PRODUCT (TYPE AND MMOUNT): Kempon 150	2375
Kempon 60	1589
N-32 yellows (for 11-60)	4
N-32 (Extrato)	

	Dec. 10,176
PRODUCT A	LBS.
PORT:	9912-16-09
SAL:	9000
DAXAD:	
MURIATIC ACID:	4020
598 OIL:	240 €
SILICA:	36001
ICE:	400
DRIMS (N-32 CLEAR):	33
FINISHED PRODUCT:	6600
KEMPORE	
UREA:	13000
MONOHYDRAZINE:	9660
SULFURIC ACID:	17836
DAXAD:	28
BICHROMATE:	9300/
ANTIFOAM:	4/
Her Plant Hydrozine 1800 GALS	1681
DRUMS (W-55 FIBER):	2/
DRUMS (N-32 YELLOW):	46/
BOXES AND LINERS:	
KEMPORE 200:	300
PINISHED PRODUCT (TYPE AND MACUNT): Kemon	
W'55's (Vard for Arunning 201 de	ne) 18

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PRODUCT A	LBS. 16 1963
PORT:	165000
SAL: DAXAD:	225001
MURIATIC ACID:	100501
598 OIL:	600/
SILICA:	7200
ICE:	100/
DRUMS (N-32 CLEAR):	66 V
FINISHED PRODUCT:	/3200
EMPORE	
UREA:	13200
HONCHYDRAZINE:	9660
SULFURIC ACID:	17681
DAXAD:	27 /
BICHRCMATE:	9500
ANTIFOAM:	8 /
Plant Hydragin 1800	1949
DRUMS (W-55 FIRER):	1
DRUMS (N-32 YELLOW):	25.
BOXES AND LINERS:	
KEMPORE 200:	150
FINISHED PRODUCT (TYPE AND RHOUNT): K	empor 125 3125
	· · · · · · · · · · · · · · · · · · ·
N-32's Yellow Kempore 60	16.
Kompore 60	2000
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- 4 -	Pec. 27 1763
PRODUCT A	LBS.
PORT:	3300
SAL:	4500
DAXAD:	
MURIATIC ACID:	5010
598 OIL:	1201
SILICA:	1500
ICE:	21/
DRUMS (N-32 CLEAR):	12
FINISHED PRODUCT:	2400
KEMPORE	
UREA:	6500
MONOHYDRAZINE:	4830
SULFURIC ACID:	12031
DAXAD:	18/
BICHROMATE:	6300 :
ANTIFOAM:	
Her Plant Hydragina 1800 calo	2179,
DRUMS (W-55 FIBER):	3 🗸
DRUMS (N-32 YELLOW):	32 /
BOXES AND LINERS:	
KEMPORE 200:	450
FINISHED PRODUCT (TYPE AND MMOUNT): Kempon 150	4000
Kempon 60	1375
,	

as <u>]</u> =	0
	Dec. 30 1763
PRODUCT A	LBS.
PORT:	16500
SAL:	22500/
DAXAD:	
MURIATIC ACID:	10,050
598 OIL:	600/
SILICA:	7300/
ICE:	99 V
DRUMS (N-32 CLEAR):	66 /
FINISHED PRODUCT:	13200
KEMPORE	
UREA:	13000
MONOHYDRAZINE:	9660
SULFURIC ACID:	19426
DAXAD:	33 /
BICHROMATE:	10 900 - :
ANTIFOAM:	<u></u>
sor: Plant Hydraine 3000 gols	3534
DRUMS (W-55 FIRER):	
DRUMS (N-32 YELLOW): K-130 = 8	28 14 V
BOXES AND LINERS:	
KEMPORE 200:	450 150
FINISHED PRODUCT (TYPE AND MAOUNT): Kampu 150	1600
Kemper 125	750
N'32 yellows drums (FOR Kempon 60)	12 🗸
Kempon 6	
16 mpou 6	1500

NATIONAL POLYCHEMICALS INC WILMINGTON MASSACHUSETTS 01887

telephone [and] als 104 to excite in the capies NAPLL ACBURNMASS

DATE: December 18, 1967.

TO: NEP cc: CPR. HRL

FROM: ARG

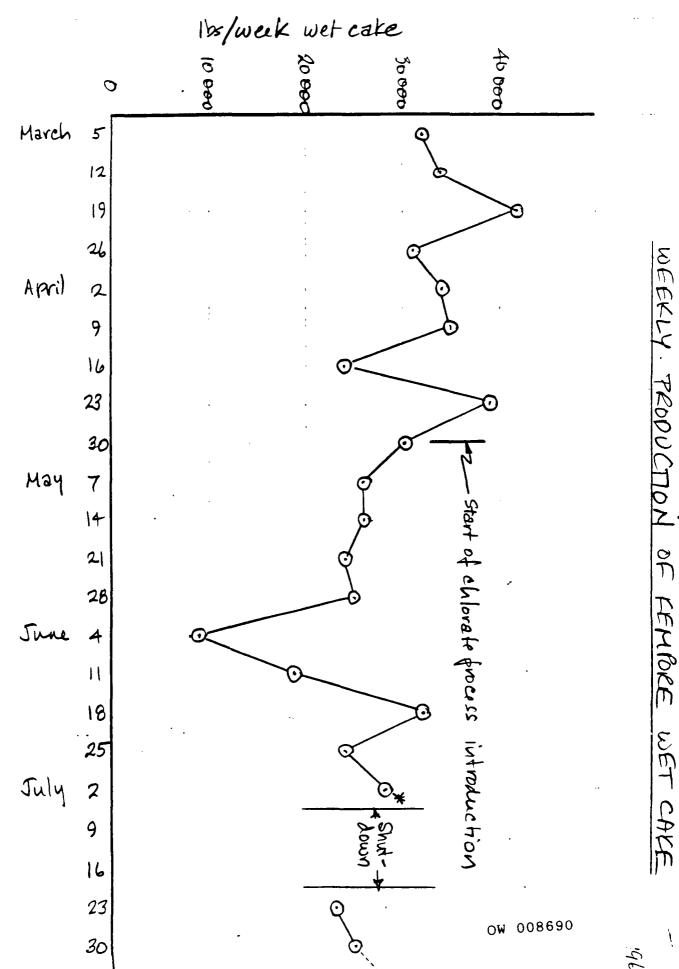
SUBJECT: KEMPORE ECONOMICS: Your memo 12/7/67

The following are my best estimates for 1968 raw material prices

	Basis	per 1b. del. ,0 77
Urea	\$78.00 per ton delivered	.0390 <u>.074</u>
Chlorine	\$69.00 per ton less 123% plus .575 freight	.03574
Sodium hydroxide	\$61.00 per ton del. on 769 Na ₂ 0 basis. May well get \$60.00 (=\$.00 at 31.5% NaOH)	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,
Sulfuric acid	\$31.15 per ton plus 8# TC freight	rate .0164
Sodium chlorate	Negotiated price for 100 lb. drums	.101
Sodium bromide	Pfizer material in drums	.42
Sodium sulfite		.0645
DOP	50% at .17 50% at .165	.1675



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tim	124 -13	31	3120
- Fels	2002	27	4500
i7: such	37, 725	<i>3</i> 1	4450
April	115, 372	3 =	3830 Reduced Production
4 iay	91, 585	30	3100
fre	71, 515	2 ፤	3280 - AI
- tuly	47, 710	15	3200 Choparte
Aug	101, 775	31	3550
jept.	93, 475	2 ق	3340] Now Filter
- 00	118, 694	31	3940
Nov	115,300	ما 2	4420 Total > 1,168,346
· jec.		27	(11 mos.)
•	1966		
mat m	65,666	۷	2744 6 Day
Feb	68,025	24	1840
Misret	135, 858	31	4360 .211
- Idpml	159, 988	30	5340 dechromote
May	69,043	3=	5640 7 dray
= tre	61,500	28	4520
tuly	87,050	(<i>5</i>	7050
Aud	131,743	31	4240
- Feps	138,482	۲8	4930
20	87, 608	3 l	2810
- Nov	80,402	Lie	3-70
Dec	112,715	บา	9200 OW 008694

total -> 1 337 44

MAPORE OXIDATION ("F" REACTOR)

Because of the increasing number of Kempore oxidation batches which are being lost because they are "blown" out of the reactor, the following procedure will followed without exception even though the batch time will be increased.

- (1) On batches which have been thickened for 3 hours, add sodium chlorate until 110-115 gallons are in the reactor.
- (2) Stop adding sodium chorate selution.
- (3) After sodium chlorate is stopped, allow batch to mix for 10 minutes.
- (4) Take a sample of the slurry at the end of the 10 minute mix and rem a DMSO completion.
- (5) If there is a relatively large amount of HDCA present, add 14 gallens more of sodium chlorate solution, mix for 10 minutes and sample for DESO completion. When the amount of HDCA becomes relatively small, add only 7 gallons of sodium chlorate, mix 10 minutes and sample for DESO completion.
- (6) By using this method it is possible to complete a batch without losing it or without observing any visual signs of completion such as forming, furing, rising in the reacter, etc.

THE COLUMN IS TO COMPLETE THE KENPORE OKIDATION WITHOUT LOSING ART OF THE PERCENT AND WITHOUT USING EXCESS SCOTING CHLORAGE STREET THE EXCESS DESTROYS THE EXMPORE.

Inventors: CHALLINOR JOHN MINTON (N/A); PORTER NEIL WILLIAM WOODROW (N/A)

Assignee: WHIFFEN & SONS LTD GB Assignee Code: 91984

Patent Issue Number Date

Patent:

US 3366622 680130

(Cited in 003 later patents)

Equivalent Patents: NL 6510442

Exemplary Claim:

A PROCESS FOR THE OXIDATION OF AN HYDRAZOAMIDE, SUCH AS HYDRAZOFORMAMIDE TO

AZOFORMAMIDE, IN HIGH YIELD WITHOUT CONTAMINATION BY THE STARTLING MATERIAL, WHICH PROCESS COMPRISES OXIDIZING THE HYDRAZOAMIDE WITH A WATER-SOLUBLE CHLORATE IN AN AQUEOUS ACRIDIC MEDIUM HAVING A PH IN THE RANGE OF 2.0 TO 0.5 IN THE PRESENCE OF A CATALYTIC AMOUNT OF BROMIDE OR BROMINE.

CA Ref: 65003756 Class: 534586000

Class Cross Ref: 534886000; 564035000

S3 1 PN="2988545"

?ts3/5/1

3/5/1

DIALOG(R)File 340:CLAIMS(R)/US Patents Abs (c) 1994 IFI/Plenum Data Corp. All rts. reserv.

0096117 6104597

C/ MANUFACTURE OF AZODICARBONAMIDE

Document Type: UTILITY
Inventors: HILL HENRY A (N/A)

Assignee: NATIONAL POLYCHEMICALS INC Assignee Code: 58560

Patent Issue Number Date

Patent:

US 2988545 610613

(Cited in 007 later patents)

Exemplary Claim:

1. THE PROCESS OF PRODUCING AZODICARBONAMIDE, WHICH COMPRISES SUSPENDING APPROXIMATELY ONE PART, BY WEIGHT, OF HYDRAZODICARBONAMIDE IN TWO TO FOUR PARTS, BY WEIGHT, OF WATER TO FORM AN AQUEOUS SLURRY, ADDING TO SAID SLURRY AN ALKALI METAL CHROMATE IN AN AMOUNT AT LEAST SUFFICIENT TO EFFECT COMPLETE OXIDATION OF THE HYDRAZODICARBONAMIDE, SLOWLY ADDING DILUTE SULFURIC ACID TO SAID SLURRY IN AN AMOUNT SUFFICIENT TO LIBERATE THE CRO3 FROM SAID CHROMATE AND TO EFFECT OXIDATION OF THE HYDRAZODICARBONAMIDE TO PARTIALLY CONVERT THE HYDRAZODICARBONAMIDE

AZODICARBONAMIDE WHILE MAINTAINING THE REACTION MIXTURE AT A TEMPERATURE

OF APPROXIMATELY 85° F., THEREAFTER HEATING THE REACTION MIXTURE TO A

TEMPERATURE BETWEEN 110* AND 150* F. UNTIL APPROXIMATELY COMPLETE CONVERSION TO AZODICARBONAMIDE HAS BEEN ACHIEVED, AND FINALLY FILTERING, WASHING AND DRYING THE AZODICARBONAMIDE THUS PRODUCED.

CA Ref: 55026496 Class: 534586000

Class Cross Ref: 534886000

THIS AGREEMENT dated this 17th day of February, 1964, between American Biltrite Rubber Co., Inc., a Delaware corporation, having its principal place of business in Chelsea, Massachusetts (hereinafter called the "Seller") and NATIONAL POLYCHEMICALS, INC. a Massachusetts corporation having its principal place of business in Wilmington, Massachusetts and formerly named "Whiffens, Inc." (hereinafter called the "Buyer").

WITNESSETH THAT:

The Buyer desires to purchase substantially all of the assets formerly owned by the Seller's wholly owned subsidiary, National Polychemicals, Inc., a Massachusetts corporation, and to assume certain of its liabilities and the Seller desires to sell such assets subject to such liabilities to the Buyer, and to that end the Buyer and the Seller do hereby represent, warrant, covenant and agree as follows:

Article One. Liquidation of National. On January 24, 1964, the Seller liquidated said National Polychemicals, Inc., and thereupon vested in the Seller title to all of the assets of said National Polychemicals, Inc. of every name, nature and description and wherever located and since said liquidation the Seller has operated the business formerly conducted by said National Polychemicals, Inc. as a separate division, known as "The National Polychemicals Division". Any reference herein to "National" shall be deemed a reference to said

National Polychemicals, Inc. or The National Polychemicals Division of the Seller, as the case may be.

Article Two. Assets to be Purchased and Sold. The Buyer agrees to purchase and the Seller agrees to sell, convey, assign, transfer and deliver to the Buyer all of the property owned and used by National in connection with the operation of its business in the fields of activity described in Article Eleven hereof, which business is principally conducted from the Seller's premises located in Wilmington, Massachusetts and Conway, North Carolina. The property to be sold and purchased shall include, without limitation, all of National's real, personal, tangible, intangible or mixed property and assets wherever located, franchises (except National's corporate franchise) contracts, good will, business as a going concern and the right to use the corporate name "National Polychemicals, Inc." and any and all trade names, trademarks, trademark applications, patents, patent applications, copyrights, trade secrets, secret processes, know-how and any developments, improvements, ideas or inventions and disclosures, whether patentable or not, conceived, disclosed, or actually or constructively reduced to practice prior to the Closing Date and the like, except for the following assets existing on the Closing Date (as hereinafter defined) which assets are to be retained by the Seller:

SUMMARY OF U. S. PATENTS OF MATIONAL POLYCHEMICALS, INC.

PATENT NO.	DATE ISSUED	TITLE AND INTENTOR
2,988,545	6/13/61	Manufacture of Azodicarbonamide Henry A. Hill
3,121,066	2/11/64	Nitroso Glycolurils William P. terHorst

THIS LICENCE AGREEMENT is made this CHI day of October 1968
BETWEEN FISONS INDUSTRIAL CHEMICALS LIMITED, formerly called WHIFFEN &
SONS LIMITED, a British Company, of Willow Works, Derby Road, Loughborough,
Leicestershire (hereinafter called "the Licensor") of the one part and
NATIONAL POLYCHEMICALS INC., a corporation of the Commonwealth of
Massachusetts, of Eames Street, Wilmington, Massachusetts (hereinafter
called "the Licensee") of the other part

NHEREAS the Licensor is the proprietor of United States Patent.

Number 3366622 dated January 30, 1968 and entitled "Oxidation of hydrazoanides to azoformmides" hereinafter called "the said patent"

AND WHEREAS the Licensee desires to obtain and the Licensor is willing to grant the licence hereinafter provided

NOW THEREFORE for and in consideration of the respective premises and covenants herein contained the Licensor and Licensee do hereby agree as follows:-

- 1) The Licensor hereby grants to the Licensee a licence to make use and sell the invention in accordance with the claims of the said patent
- 2) The Licence is royalty free and subsists as long as the said patent or any extensions or reissues thereof is in force.
- 3) The Licence is personal to the Licensee and is not assignable and does not include the right to grant sub-licences.
- 4) The Licence is non-exclusive.
- 5) The Licensee agrees not to dispute the validity of the claims of the said patent while this Agreement is in force.
- 6) The Licence granted by this Agreement is limited to the said patent.

IM WITNESS WEEREOF these presents have been duly executed as of the day and year first before written.

The COMMON SEAL of FISONS
INDUSTRIAL CHEMICALS LIMITED
was hereunto affixed in the
presence of:-

Dhes.

NATIONAL POLYCHEMICALS INC

Ву

C00074

Revised: July 1, 1969

II. OXIDATION TO HECE BY CHLORATE

A. All chlorate exidations should be run in the "F" reactor or the "C" reactor in an emergency. The batch should be charged as follows:

- 1. Check all valves at the rubber-lined held tank, polyester tank, transfer pump suction and discharge, and reactor, to be sure they are in the proper position for rumping from the polyester tank to "F" reactor. Be sure that chlorate valves at reactor are closed.
- 2. Charge thickened HDCA clurry from polyester hold tank to "F" reactor. Before charging, if headspace in polyester tank is more than 75", bring batch up to 1,000 gallons by charging unthickened HDCA from rubber-lined hold tank to polyester tank to a final headspace of 75"; and pump entire batch to "F" reactor. Flush all transfer lines with water after pumping to "F" reactor.
- 5. Charge 200 gallons of sulfuric acid through the acid mater while transferring the thickened HDCA. If the acid mater is incorrative, the MDCA should be pumped and then 8° of acid added. It is extremely important to monitor the temperature of the reactor; the final temperature after acid addition should be no lower than 120° F. and can be controlled by cooling water on the internal heat exchangers.
- 4. Charge 5 lbs. of solid bromide to reactor.
- 5. Prepare 2 pails of sodium sulfite for end of batch. For each pail, dissolve 5 lbs, of sulfite in 4 gallons of hot water, with continuous stirring. Additional dry culfite in bulk should be on hand to be used if neutralization requires more.
- 6. After batch has been charged, thickening should be started immediately on the name batch. See section II for detailed procedure.

E. Chlorats Addition

- 1. Record chlorate tank headopace before starting chlorate addition. The chlorate tank agitator should be running at all times when there is solution in the tank, except when reading headspace.
- 2: Nike sure that enforces values at reactor are closed; over value in chlorate tank recycle line, and start chlorate punt. Pe sure that tecycle line is discharging. In it is not, clean out arrainer in chlorate punc suction line; and restart pump.
- 3. Chock the internal batch temperature; and, if it is above 130° F., turn on cooling water to reactor jacket.

Revisea: July 1, 1969

4. Start chlorate addition and control rate by throttling valve at reactor. Batch will probably show some foaming initially. With a flow of approximately 1.0 gpm, millivolts indicated on recorder should rise to 750 to 780. After about five minutes, batch should begin to appear yellowish. If no color change is noted, it indicates that the catalyst (bromide) was omitted; and 5 lbs. of bromide should be added.

NOTE: ALL NORMAL BATCHES ARE OXIDIZED AT 130 - 135° F., WITH THE EXCEPTION OF AN OCCASIONAL SPECIAL SERIES OF BATCHES WHICH ARE OXIDIZED AT 140 - 145° F. THIS OXIDATION TEMPERATURE IS EXTREMELY IMPORTANT, BECAUSE IT AFFECTS FILTER OPERATION.

- 5. After initial foaming subsides, increase the chlorate addition rate. The millivolt reading should adjust to the range of 790 to 815. There is no precise millivolt reading to be aimed for. It is more important to get a constant reading with a constant chlorate flow rate and constant temperature.
- 6. While running, it is very important to maintain the batch temperature in the range of 130° to 135° F. Temperature readings should be taken at least every 5 minutes and the cooling water flow adjusted as required. A low temperature will give a slow reaction rate, and thus a higher excess chlorate and millivolt reading, for any given chlorate flow, than will a high temperature. If, at any time, the temperature drops below 130° F., cooling water should be turned off until temperature comes back up. If millivolts are above 815 because of low temperature, the chlorate rate should be slowed down to reduce excess chlorate and batch allowed to react slowly until temperature comes back up. If batch temperature rises above 1950 Per chlorate flow should be turned off and cooling water turned on full until temperature comes down to 130° F.
- 7. The amount of chlorate which has been added should be closely followed by checking chlorate tank headspace at least every 15 minutes. One inch of headspace equals 14 gallons. If batch is to be unattended, the chlorate flow should be shut off while checking headspace. Unless temperature is ever 1950 F., cooling water should be turned off whenever chlorate is off in order to keep batch temperature from dropping.
- 6. The progress of the batch can be checked at any time by running DNSO completions, as detailed in Section C III. Be sure to use hot water wash at end to check white residue for salt. In any case, a completion should be run after 110 to 115 gallons have been added, and as frequently as required after that.

Revised: July 1, 1969

9. At any time that there are any questions or problems about the progress of a batch, the chlorate can be shut off without doing any damage. Be sure that cooling water is also off, unless temperature is high, so that batch will not cool below 130° F.

10. An operator should be at the reactor menhole at all times when chlorate flow is on. If batch is to be unattended for even a very short time, chlorate and cooling water flows should be turned off.

C. End Foint Determination

- 1. The batch color gives a rough indication of progress, from a creemy yellow near the beginning to a dark yellow-crange as the erd point nears. The disappearance of white foam near the agitator shaft also indicates very near completion. Excessive foaming or red fumes are also an indication of batch completion.
- 2. The redox meter is <u>not a reliable method</u> of determining the endpoint based upon past experience; the best method is the DMSO completion test (See Section D). The endpoint of each batch should be determined as follows:
 - (a) On batches which have been thickened for 3 hours, add sodium chlorate until 110 115 gallons are in the reactor.
 - (b) Stop adding sodium chlcrate sclution.
 - (c) After sodium chlorate is stopped, allow batch to mix for 10 minutes.
 - (d) Take a sample of the slurry at the end of the 10 minutes mix and run a DMSO completion.
 - (e) If there is a relatively large amount of HDCA present, add the gallons more of sodium chlorate solution, mix for 10 minutes and sample for DMSO completion. When the amount of HDCA becomes relatively small, add only 7 gallons of sodium chlorate, mix 10 minutes and sample for DMSO completion.

NOTE: THE WHITE RESIDUE SHOULD BE CHECKED FOR SOLUBILITY IN WARM WATER TO DETERMINE THAT IT IS NOT SALT.

(f) By using this method, it is possible to complete a batch without losing it or without observing any visual signs of completion such as foaming, fuming, rising in the reactor, etc.

Revised: July 1, 1969

3. The redox meter can be used as a <u>paneral</u> indicator of completion. A rapid rise in millivelt reading will normally indicate completion; the chlorate should be stopped, the batch allowed to mix for 10 minutes, and then sampled for the EMSO completion test.

MOTE: IT IS EXTMENDLY EMPORTANT TO ADD CHLORATE SLOWLY WHEN APPROACHING COMPLETION SO AS NOT TO ADD A LARGE ENGASS OF CHLORATE, AND IT IS ALSO IMPORTANT NOT TO LET TEMPERATURE DEEP AS REACTION RATE WILL FAIL OF DRASTIGALLY.

4. When the completion is confirmed by DMSO test, add both pails of sulfite solution. Additional solid sulfite may be added if necessary; but, in no case should a total of more than 40 lbs. be added.

NOTE: IF THERE IS ANY DOUBT, THE SULFITE SHOULD BE ADDED AS IT IS BETTER TO ADD SULFITE TOO SOON THAN TOO LATE. THE SULFITE WILL NOT DESTROY THE BROMIDE CATALYST, BUT ONLY PREVENT IT FROM BEING RELEASED AS FREE BROMINE VAPOE. IF SULFITE IS ADDED BEFORE BATCH IS COMPLETE, THE BATCH CAN STILL BE CONTINUED BY ADDING ADDITIONAL CHIORATE.

5. After adding sulfite, the batch should again be checked for completion with DMSO. If completion test is good, the cooling water should be turned on and the batch diluted with 200 gallons of cold water. Start pumping to the filter feed tenk immediately. Do not cool the batch to a specific temperature range or wait for the entire 200 gallons of water to be added.

D. HDCI Corpletion Test

- 1. Take sample of batch, filter, and wash.
- 2. Add $\frac{1}{2}$ " of filtered and washed product to clean test tube.
- 3. Fill test tube three-quarters full with DMSO, shake well, contribuge, and drain the liquid from the test tube.
- 4. Repeat Section 3 two additional times. A white recidue after the third centrifucing indicates the presence of unreacted HDCA; i.e., batch is not complete. Before assuming that the batch is not complete, the residue should be carefully checked for solubility in warm water to be sure that it is not salt.

TENTATIVE MANUFACTURING PROCEDURE

HDCI

	Lbs.	Density	<u>Gallons</u>
HDCA	2,890 (dry)		1,000
Sulfuric Acid	3,375	15.0	225
Sodium Chlorate	1,104 (dry)	11.0	252
Sodium Bromide	10		
Sdoium Sulfite	20		
Antifoam	2		

- (1) Pump 1000 gallons of completed HDCA slurry (72" headspace as measured from the top of the manhole) to the "F" reactor.
- (2) Charge 225 gallons of sulfuric acid to the reactor. Cooling water should be used to keep the mixture below 120° F. during the acid addition.
- (3) Add 10 lbs. sodium bromide.
- (4) Start charging 40% sodium chlorate solution while maintaining the temperature of the mixture at 120 125° F. (48.9-51.7°c)

NOTE: This temperature range is extremely critical since it governs particle size of the finished product. If the oxidation is carried out above this range, the particle size will be too large to grind to K-125 on one pass; if the reaction is carried out at too low a temperature, the material will be difficult to filter.

(5) Sample periodically for reaction completion using DMSO.

Effect MOETA Oct. 8, 8/

3/28/78

Kempore (azodicarbonamide or 1, 1' azobisformamide)

I. Two Stage Reaction

O.C. 1. 12. 2-11.05

Safety: 41 1/45

MT 11 1. may 4/45

RMG mld 1 1/1/145

OLIN CORPORATION MANUFACTURING PROCEDURE KEMPORE

I. Batch Charge

HDCA

Hydrazine (65%) = 660 gals (sp.gr. = 8.61 lbs/gal)

Urea 150 bags = 15,000 lbs Water 1201 gals = 10,016 lbs

Sulfuric Acid 260-300 gals 3,900 - 4,500 lbs (sp.gr. = 15.3 lbs/gal)

HDCI

#DCA 952 gals = 2,676 lbs (dry)
Sulfuric Acid approx.200 gals= determined by Q.C.

Sodium Chlorate 175-350 gals= determined by Q.C. (40% approx.)

Sodium Bromide = 10 lbs
Sodium Sulfite = 10-20 lbs
Anti foam = as needed

"II. Finished Product Specifications

1. Appearance yellow color

2. Decomposition point 190°Cminimum (374°F)

3. Moisture 0.3% maximum
4. HDCA content 0.1% maximum
5. Residue on 325 mesh screen 0.01% maximum

6. ASH 0.2% maximum 0.2% maximum

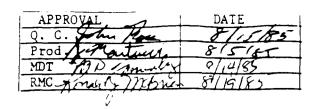
II. For HDCA Reaction (Step I) In S.S. Reactor

A. Hexamine Scrubber

(A) Formaldehyde, 50% approx. 1,750 gals
Hexamine 32% 2,300 gals

B. Ammonia Scrubber

(A) Water . 1,500 gals
Aqua ammonia yield . 1,546 gals



Replaces: November, 13

Page 1 of 13

OLIN CORPORATION MANUFACTURING PROCEDURE

KEMPORE

I. BATCH CHARGE

HDCA

Hydrazine (65%) = 660 gals (sp.gr. = 8.61 lbs/gal)

Urea 150 bags = 15,000 lbs or 300 bags @ .50c = 15,000#

Water 1201 gals = 10,016 lbs

Sulfuric Acid 260 - 300 gals = 3,900 - 4,500 lbs (sp.gr. = 15.3 lbs/gal

HDCI

HDCA 952 gals = 2,676 lbs (dry)
Sulfuric Acid approx. 200 gals = determined by Q.C.
Sodium Chlorate 175 - 350 gals = determined by Q.C.

Sodium Bromide = 10 lbs
Sodium Sulfite = 10 - 20 lbs
Anti foam = as needed

II. Finished Product Specifications

1. Appearance yellow color

2. Decomposition point 190°C minimum (374°F)

3. Moisture
4. HDCA content
5. Residue on 325 mesh screen
6. Ash
0.2% maximum
0.2% maximum

III. For HDCA Reaction (Step I) in S.S. Reactor

A. Hexamine Scrubber

(a) Formaldehyde, 50% Approx. 1,750 gals Hexamine 32% 2,300 gals

B. Ammonia Scrubber

(a) Water 1,500 gals
Aqua ammonia yield 1,546 gals

Revised: January 1986 Replaces: July 1985

APPROVAL; DATE:

Q.C. John Place 3/5/86

Prod. Jon Martiner 3/12/86

MDT Mil murley 3/12/86

RMc Aprild McFrie 3/13/86

OLIN CORPORATION

MANUFACTURING PROCEDURE

KEMPORE

BATCH CHARGE

١.

HDCA (per S.S. batch)

	<u>Pounds</u>	Gallons	<u>Density</u>	
Hydrazine (65%)	5,680	660	8.61 lb/g	ja l
Urea	15,000			
Water	10,016	1,201	8.34 lb/g	ga I

HDCI (per batch)

	<u>Pounds</u>	<u>Gallons</u>	<u>Density</u>	
HDCA	2,676 (dry)	952		
Sulfuric Acid	2,907	190	15.30	lb/gal
Sodium Chlorate	1925-3850	175 - 350	11.00	lb/gal
Sodium Bramide	10			
Sodium Sulfite	5-10			
Antifoam	as needed 5 max.			

continued-

I, Maurice J. Bernstein, President of American Biltrite Rubber Co., Inc., a Delaware corporation, hereby certify that attached hereto is the Plan of Liquidation of National Polychemicals, Inc. as suggested to it by this company as sole stockholder.

Given under my hand and the seal of the corporation this 24th day of January, 1964.

AMERICAN BILTRITE RUBBER CO., INC. (Delaware) Chelsea, Massachusetts

To the Stockholders and Directors of National Polychemicals, Inc. (Massachusetts)

The undersigned, being the owner of all of the stock of your corporation, proposed the following Plan of Complete Liquidation of National Polychemicals, Inc. (Massachusetts).

- 1. On January 24, 1964, as of the close of business, National Polychemicals, Inc. will transfer and distribute all of its assets and property, including its name and goodwill, to American Biltrite Rubber Co., Inc. (Delaware) ("Biltrite") in complete liquidation of National Polychemicals, Inc. and in consideration of (a) the assumption by Biltrite of all debts and obligations of National Polychemicals, Inc. to third persons, whether such debts and obligations are liquidated, contingent or arise hereafter, and (b) the surrender to National Polychemicals, Inc. by the undersigned for the complete cancellation by National Polychemicals, Inc. of all outstanding shames of capital stock of National Polychemicals, Inc.
- It is intended that the Complete Liquidation of National Polychemicals, Inc. in accordance with the Plan shall be tax free under the provisions of Section 332 of the Internal Revenue Code of 1954.

AMERICAN BILTRITE RUBBER CO., INC. (Delaware)

1 // -live

Dated: January 24, 1964

BILL OF SALE AND GENERAL CONVEYANCE

INDENTURE dated January 24, 1964 between NATIONAL PCLYCHEMICALS, INC., a Massachusetts corporation, hereinafter sometimes called "NATIONAL" and AMERICAN BILTRITE RUBBER CO., INC., a Delaware corporation, hereinafter sometimes called "BILTRITE".

WHEREAS Biltrite owns all of the issued and outstanding shares of stock of National; and

WHEREAS National, by votes duly adopted by its Board of Directors and by votes unanimously adopted by all its stock-holders at meetings duly called and held on January 24, 1964, has approved and adopted a Plan of Complete Liquidation of National, which Plan has also been approved and adopted by Biltrite: and

whereas under said Plan, all of the assets and property of National, including its name and goodwill, are to be transferred and distributed to Biltrite, in complete liquidation of National and in consideration of (a) the assumption by Biltrite of all debts and obligations of National to third persons, whether such debts and obligations are liquidated, contingent or arise hereafter, and (b) the surrender to National by Biltrite for complete cancellation by National of all outstanding shares of capital stock of National;

NOW, THEREFORE, in consideration of the premises and for value received, the receipt whereof is hereby acknowledged. NATIONAL POLYCHEMICALS, INC. (National"), a Massachusetts corpcration, does hereby give, grant, bargain, sell, assign, transfer, convey, set over, release and confirm unto American Biltrite Rubber Co., Inc., a Delaware corporation, its successors and assigns, all the property and assets owned of record or otherwise by National of every kind, nature and description, real, personal and mixed, and wheresoever situated, including, but without limiting the generality of the foregoing grant, its business as a going concern, the right to its corporate name, its goodwill, trademarks, trade names, franchises, copyrights, patents, patent rights, applications for patents, inventions and improvements, processes, licenses, license agreements, lands and interests in lands, whether in fee or of less estate, purchase options, water and riparian rights, plants, factories, mills, buildings, machinery, tools, dies and patterns, vehicles and equipment, inventory, merchandise, raw, finished or in process, supplies, tangible personal property of all kinds, bonds, notes and other obligations, choses in action, claims, rights, causes of action, judgments, bills and notes receivable, accounts receivable, claims for refund or abatement of taxes,

claims for tax relief, rights to recover in tort, contract or otherwise, contracts, guarantees, deeds, leases, mortgages, assignments, orders, cash, bank accounts, policies of insurance, all equitable interest in property whether standing in the name of National, or in the name of any other person for the use and benefit of National, and all other intangibles, books of account, files, papers and records; excepting from this conveyance any items which by their nature are non-assignable, the proceeds of which shall be transferred and conveyed to Biltrite upon receipt by National.

TO HAVE AND TO HOLD the same, with all the appurtenances thereto, unto Biltrite, its successors and assigns forever, to its and their own proper use and behoof forever, but without contracts of warranty, express or implied.

National hereby constitutes and appoints Biltrite, its successors and assigns, the true and lawful attorney or attorneys of National, with full power of substitution, in the name of National or otherwise, but on behalf of and for the benefit of Biltrite, its successors and assigns, and at the sole cost and expense of Biltrite, or its successors and assigns, to demand, enforce, sue for, collect, and receive from time to time any and all assets, property and claims, tangible or in-

tangible, hereby assigned, transferred and conveyed or intended so to be; to give receipts, releases and acquittances for or in respect of the same or any part thereof, and from time to time to institute and prosecute in the name of National or otherwise any or all proceedings at law, in equity or otherwise, which Biltrite, its successors or assigns, may deem advisable; to collect, assert or enforce any claim, right, title, interest, deed or account hereby assigned, transferred or conveyed or intended so to be; to defend and compromise any and all actions, suits or proceedings in respect of any of the assets, property and claims hereby assigned, transferred and conveyed or intended so to be; and to do any and all acts and things in relation thereto that Biltrite, its successors or assigns, shall deem desirable; National hereby declaring that the foregoing powers are coupled with an interest and shall be irrevocable. National hereby agrees that any and all claims, property or assets which by their nature are non-assignable shall be held, together with all proceeds thereof received or realized by it, in trust for Biltrite and upon receipt will be forthwith by Mational paid, transferred, assigned, turned over and delivered to Biltrite.

Biltrite hereby assumes and agrees to pay, perform and

discharge as they severally become due all debts and obligations of National to third persons and all contracts of National, whether such debts and obligations are liquidated, contingent or arising after the date hereof.

National hereby acknowledges receipt from Biltrite of all National's outstanding shares of capital stock, such shares having been surrendered by Biltrite to National for complete cancellation by National.

National hereby further covenants and agrees that it will execute and deliver to Biltrite, its successors and assigns, such other and further assignments, transfers, conveyances, confirmations and other instruments of further assurances, approvals and consents, whether by itself or others, as may be considered by Biltrite, its successors or assigns, to be necessary or advisable to complete, insure, confirm or protect the transfer to Biltrite, its successors or assigns, of all the right, title and interest of National in and to any and all of the assets, property and claims hereby assigned, transferred or conveyed or intended so to be.

This Indenture may be simultaneously executed in several counterparts, each of which so executed shall be deemed to be an original, and such counterparts together shall constitute one and the same instrument.

IN WITNESS WHEREOF, the parties hereto have caused this Indenture to be executed and their corporate seals to be affixed by their duly authorized officers as of the day and year first above written.

NATIONAL POLYCHEMICALS, INC. (Massachusetts)

Treasure:

AMERICAN BILTRITE RUBBER CO., INC.

(Delaware)

y: Manue / Dennie

Commonwealth of Massachusetts) County of Suffolk

On this 24th day of January, 1964, before me personally came Aaron J. Bronstein, to me personally known, who, being by me duly sworn, did depose and say that he resides at Swampscott, Massachusetts, that he is the Treasurer of National Polychemicals, Inc., a Massachusetts corporation, one of the corporations described in and which executed the above instrument; that he knows the corporate seal of said corporation; that the seal affixed to the foregoing instrument is such corporate seal; that it was so affixed by order of the Board of Directors of said corporation and that he signed his name thereto by like authority.

My Commission Expires: 2/8/69

Commonwealth of Massachusetts) County of Suffolk

On this 24th day of January, 1964, before me personally came Maurice J. Bernstein, to me personally known, who, being by me duly sworn, did say that he resides in Newton, Massachusetts; that he is the President of American Biltrite Rubber Co., Inc., a Delaware corporation, one of the corporations described in and which executed the above instrument; that he knows the seal of said corporation; that the seal affixed to the foregoing instrument is such corporate seal; that it was so affixed by order of the Board of Directors of said corporation and that he signed his name thereto by like order; and that said Maurice J. Bernstein acknowledged said instrument to be his free act and deed and the free act and deed of said American Biltrite Rubber Co., Inc.

Notary Public

My Commission Expires: 2/5/67

INSTRUMENT OF GENERAL TRANSFER

Coxed Times, on January 24, 1904, American Biltrite Rubber Coxed Times, Inc. a Delaware corporation, having its principal place of business in Chelsea, Massachusetts (the "Seller") liquidated its vincily simed subsidiary, National Polychemicals, Inc., a Massachusetts corporation, having its principal place of business in Wilmington, Massachusetts, and thereupon vested in the Seller citle to all of the assets of said National Polychemicals, Inc. of every name, nature and description and wherever located; and

WAENERS, since said liquidation, the Seller has operated the business formerly conducted by said National Polychemicals, Inc. as a separate division known as "The National Polychemicals Division" ("National") and

WHEREAS, the Seller has agreed to sell, assign and transfer to hottond fludence, in. (17)
Whitens, Inc., a Massachusetts corporation having its principal place of business in Boston, Massachusetts (the "Buyer") substantially all of said assets of National.

NOW, THEREFORE, the Seller, for good and valuable consideration to it paid by the Buyer, the receipt whereof is hereby acknowledged, does hereby give, grant, bargain, sell, assign, transfer, set over and confirm unto the Buyer, its successors and assigns forever, all the

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property of National of every kind and nature wherever situated, real, personal and mixed (except such items of property as are harding Four specifically excluded) including, but without limiting in any may the tenerality of the foregoing grant, all of the percence property, whether tangible or intangible, located in, on or about Mational's premises in Wilmington, Massachusetts and Commay, North Carolina and National's business as a going concern, the right to the name, "National Polychemicals", its good will, trademarks, trademark applications, trade names, franchises, conyrights, patents, patent rights, applications for patents, inventions and improvements, trade secrets, know-how, secret processes, any develonments, improvements, ideas or inventions and disclosures whether patentable or not, conceived, disclosed, or actually or constructively reduced to practice prior to the Closing Date, land and interests in lands, whether in fee or of lesser estate, water and riparian rights, plants, factories, buildings, machinery, tools, vehicles and equipment, merchandise, raw, wrought or in process, supplies, tangible personal property of all kinds, choses in action, causes of action, judgments, accounts receivable, rights to recover in tort, contract or otherwise, contracts, orders, all equitable interests in property whether standing in the name of Mational or in the name of any other person for the use and benefit of Mational and all other intangibles, books of account, files, papers and records.

It is understood and agreed that the foregoing grant does not

OW 015150

include the following:

- (a) Mational's cash on hand or in banks:
- (b) National's accounts receivable owing from the Seller and its subsidiaries and all other accounts receivable, other than trade accounts receivable:
- (c) Machinery used in the direct manufacture of plastic and rubber hose, contracts and other elements of National's business relating to the manufacture of plastic and rubber hose;
- (d) National's interest in or rights under insurance policies, and

(e) Deposits

Title to all the property above mentioned shall pass to the Buyer as of the opening of business on February 17, 1964.

TO HAVE AND TO HOLD the same, with the appurtenances thereof, unto the Buyer, its successors and assigns forever, to its and their own proper use and behoof.

The Seller hereby constitutes the Buyer, its agent and attorney, but at the Buyer's expense, to receive, collect, enforce and sue for any of the property or rights hereby granted or assigned either in its name or in the name of the Seller or in the name of National, as the legal attorney of the Seller thereunto duly authorized for the use a the Buyer.

The Seller covenants with one Buyer that it will, and its successors and assigns shall, at any time and from time to time, at the request of the Buyer, execute and deliver to it any now or confirmatory instrument and all other and further instruments necessary or convenient or union one Buyer may request to vest in the Buyer full title, right or interest in or to any property included within the foregoing grant, or to enable the Buyer to realize upon or otherwise to enjoy any such property, or to carry into effect the intention or purpose hareof.

The Selics hereby covenants with the Buyer that it is the lawful owner of the personal property hereby transferred; that such personal property is free from encumbrances; that it has good right to sell the same as aforesaid; that it will defend the same and indemnify the Buyer against all claims and demands of all persons.

The Soller hereby covenants with the Buyer that it will assist the Buyer in the collection of the trade accounts receivable of National transferred to the Buyer hereunder, and if National should receive payment of any such accounts, it will pay over immediately to the Buyer the amounts collected.

IN WITNESS WHEREOF, American Biltrite Rubber Compeny, Inc.

be noted alfined by its only authorized officer this 17th day of Jebr 17th, 1904.

LIBRIDAN BILITAITE AUDBER COLEMA, INC.

27 Sichard Miterians

State of Delacoure

Office of the Secretary of State PAGE 1

I, EDWARD J. FREEL, SECRETARY OF STATE OF THE STATE OF DELAWARE, DO HEREBY CERTIFY THE ATTACHED IS A TRUE AND CORRECT COPY OF THE RESTATED CERTIFICATE OF "AMERICAN BILTRITE INC.", FILED IN THIS OFFICE ON THE THIRD DAY OF MAY, A.D. 1973, AT 9 O'CLOCK A.M.

OW 103852



Edward J. Freel, Secretary of State

AUTHENTICATION:

7207550

DATE:

08-10-94

0482015 8100

944149404

RESTATED CERTIFICATE OF INCORPORATION

OP

AMERICAN BILTRITE RUBBER CO. INC.

- 1. The present mame of the corporation (hereinafter called the "corporation") is AMERICAN BILTRITE RUBBER CO., INC., which is the name under which the corporation was originally incorporated; and the date of filing the original certificate of incorporation of the corporation with the Secretary of State of the State of Delaware is Movember 29, 1954.
- 2. The certificate of incorporation of the corporation is hereby amended by:
 - (a) striking out present Articles FIRST, THIRD PIFTH, SIXTH and SEVENTH is their entireties and substituting new Articles FIRST and THIRD;
 - (b) deleting subparagraphs (d), (e) and (f) of paragraph 3. of present Article SIGHTH, and deleting paragraphs 5., 6., and 7. of said Article EIGHTH, and remambering said Article, as assended, as Article FIFTH;
 - (c) striking out Article ELEVENTH in its entirety;
 - (d) remastering the present Article HINTH as Article SIXTH and the present Article TENTH as Article SEVENIH.

all of which changes are set forth in the following Restated Certificate of Incorporation.

- 3. The previsions of the certificate of insorporation of the emporation as heretofore amended and/or supplemented, and as herein amended, are hereby restated and integrated into the single instrument which is hereinafter set forth, and which is estitled Restated Certificate of Incorporation of AMERICAN BILITATE INC. without any further amendments other than the amendments herein certified and without any discrepancy between the previsions of the certificate of incorporation as heretofore amended and supplemented and the provisions of the single instrument hereisafter set forth.
- 4. The amendments and the restatement of the cortificate of incorporation herein certified have been duly adopted by the stockholders in accordance with the provisions of Section 242 and of Section 245 of the Seneral Corporation Law of the State of Delaware.

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- 5. The capital of the corporation will not be reduced under or by reason of any amendment herein certified.
- 6. The certificate of incorporation of the corporation, as amended and restated herein, shall upon the effective date of this Restated Certificate of Incorporation, read as follows: